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SUPERFUND RECORDS

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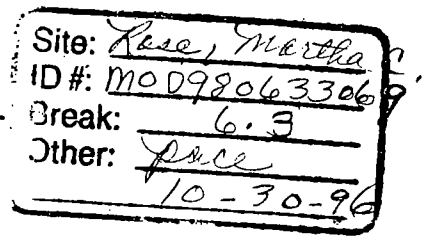
10-30-96

LABORATORY QUALITY ASSURANCE PLAN

Pace Analytical Services, Inc. - Houston Laboratory

Approvals:

Quality Assurance Officer	10/30/96 Date	Laboratory General Manager	10/30/96 Date



Pace Analytical

MARTHA
C.
Rose

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1. INTRODUCTION, PROGRAM OBJECTIVES, AND STATEMENT OF POLICY

1.1 INTRODUCTION

This Quality Assurance (QA) Plan is written in compliance with the elements required in the U.S. EPA, *Guidelines and Specifications for Preparing Quality Assurance Program Plans*, QAMS-005 80, December 1980. This document contains the required elements of a Quality Assurance Plan and is prepared in such a manner that entire sections can be referenced in subsequent specific project plans. This QA Plan shall be reviewed annually, at a minimum, by the Quality Assurance Officer and the laboratory management staff; revisions shall be incorporated as required.

The QA Plan defines the systems of quality control and quality assessment that constitute the comprehensive Quality Assurance program at the Pace Analytical Services, Inc.- Houston Laboratory (Pace-Houston). Quality control consists of specific procedures applied to all phases of analysis from sample receipt through the final reporting of results. The purpose of quality control is to ensure that quality goals are met under routine operating procedures. Quality assurance involves the continuous evaluation of data and monitoring of analytical processes for the purpose of ensuring that the quality control systems are performing effectively.

1.2 PROGRAM OBJECTIVES

The major elements of the Pace-Houston Quality Assurance Program are summarized as follow:

- Definition of the data quality objectives for the project.
- Use of appropriate methodologies by technically competent, well-trained personnel using sophisticated instrumentation and equipment to attain these objectives.
- Adherence to well-defined standard operating procedures with emphasis on good laboratory and measurement practices.
- Assessment of precision and accuracy by the use of quality control (QC) samples including, but not limited to, matrix spike samples, duplicate samples, surrogate spikes, blanks, and independent laboratory control standards.

- Use of the results of QC sample analyses for determining the measure of successful achievement of data quality objectives.

- Participation in external quality evaluation programs including the U.S. EPA Water Pollution (WP) and Water Supply (WS) Study Programs, client originated performance studies, and state agency certification programs.

- Maintenance of accreditation by state, federal, and other applicable agencies for work performed.

- Monitoring compliance with approved procedures and assessment of the performance of the analytical methods.

1.3 STATEMENT OF POLICY

Pace Analytical Services, Inc. expects from its management staff and all employees a high degree of commitment to quality assurance and to providing legally defensible data of known and appropriate quality to its clients. The validity and reliability of the information generated is optimized by the adherence to documented standard operating procedures (SOPs). Pace SOPs are written to comply the laboratory management's interpretation of the EPA-approved methods. SOPs used by Pace-Houston are also intended to comply with minimum requirements documents (MRDs), produced by Pace's corporate office to promote consistency and comparability between Pace laboratories.

Pace-Houston emphasizes the application of sound quality assurance/quality control principles beginning with the initial planning of the project, through all the field and laboratory activities and ultimately to the preparation of the final report. The principles of the data quality objectives for representativeness, completeness, comparability, precision, and accuracy are applied to the analytical data generated.

To ensure client satisfaction, Pace-Houston encourages strong interaction with the client at all phases of the project. Proactive interaction with the client assists Pace-Houston in delivering a final product that meets the project-specific data quality objectives.

Pace-Houston is committed to providing the resources, including facilities, equipment and personnel, to ensure the timely completion of analyses and adherence to applicable QA/QC protocols.

2. LABORATORY ORGANIZATION AND RESPONSIBILITY

Pace Analytical Services, Inc. operates a nationwide system of seven laboratories. Each laboratory is managed by a General Manager with responsibility for the technical and financial performance of the region. A centralized corporate staff provides support for the regional offices and coordination of inter-regional activities. This operational structure enables Pace to provide services which are responsive to the client's specific needs. It also provides a mechanism for each laboratory to utilize the total assets of the corporation to level work loads and provide a high-quality product in a timely manner.

Each laboratory is organized into departments, each headed by a Department Manager. In the Pace-Houston laboratory the technical departments are organic, inorganic, and field services. The departments of marketing and client services provides the client interface and project management staff necessary to ensure a project is completed in the manner required by the client. The department of support services provides the secretarial and accounting services necessary for operation of the laboratory. The Pace-Houston facility occupies a building of approximately 30,000 square feet; the floor plan is depicted in Figure 2-1.

Each laboratory has a Quality Assurance Officer (QAO) with responsibility for ensuring that all activities of the lab are in compliance with corporate policy for quality. The QAO reports directly to the General Manager and has the authority and the responsibility to implement and approve corrective actions as needed. The QAO is responsible for monitoring QC sample analysis results and the results obtained for analyses of external performance samples to identify potential problems. He is responsible for initiating both preventive and corrective action processes as needed to ensure proper operations within the laboratory. The QAO is also responsible for maintaining certifications required for laboratory operations.

Laboratory analysts are responsible for performing analyses according to standard operating procedures (SOPs) and for evaluating the acceptability of their data based on established quality control criteria. Analysts are also responsible for initiating corrective action when QC criteria are not met.

The organizational structure for Pace-Houston is provided in Figure 2-2. Job descriptions are on file with the laboratory personnel office for all analytical personnel. Job descriptions for specified functions will be made available upon request.

FIGURE 2-1

Pace Analytical Services, Inc. - Houston Laboratory Floor Plan

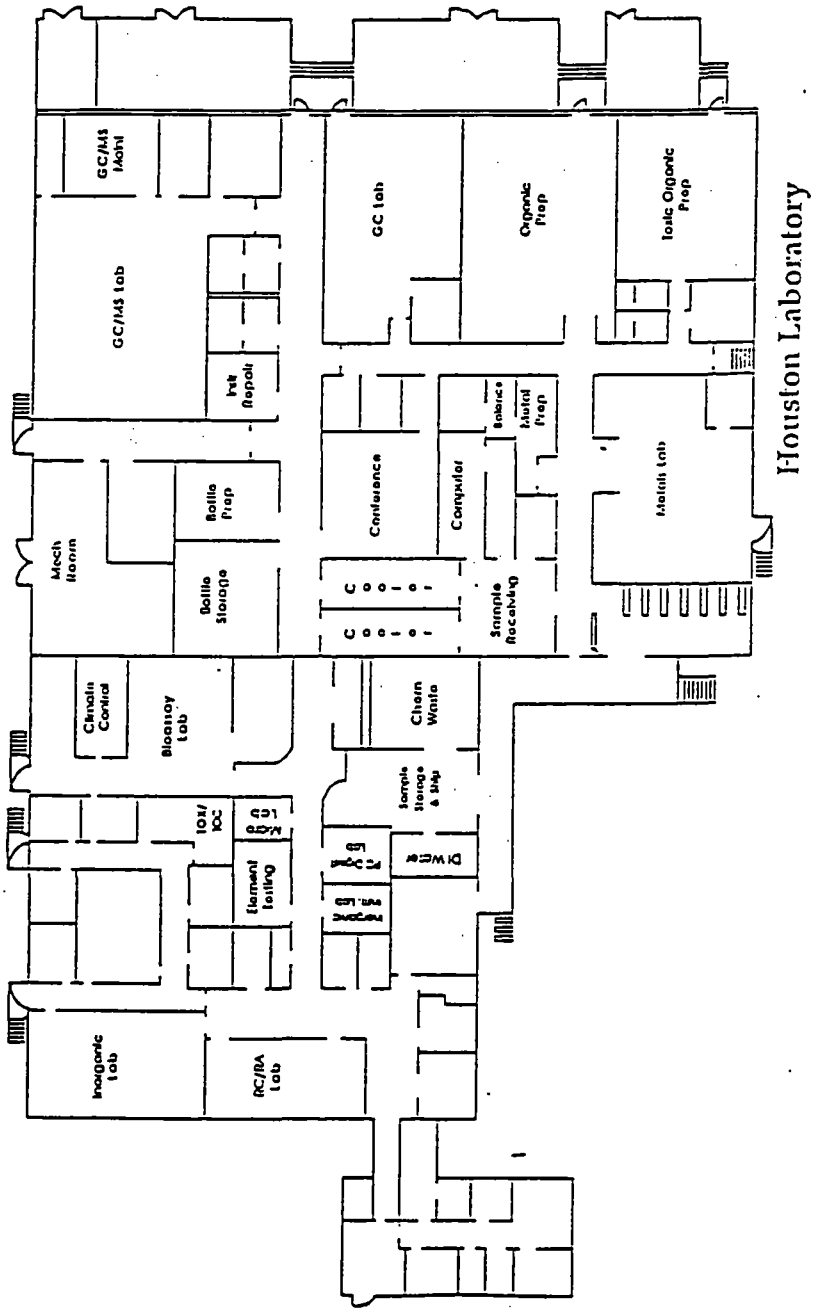
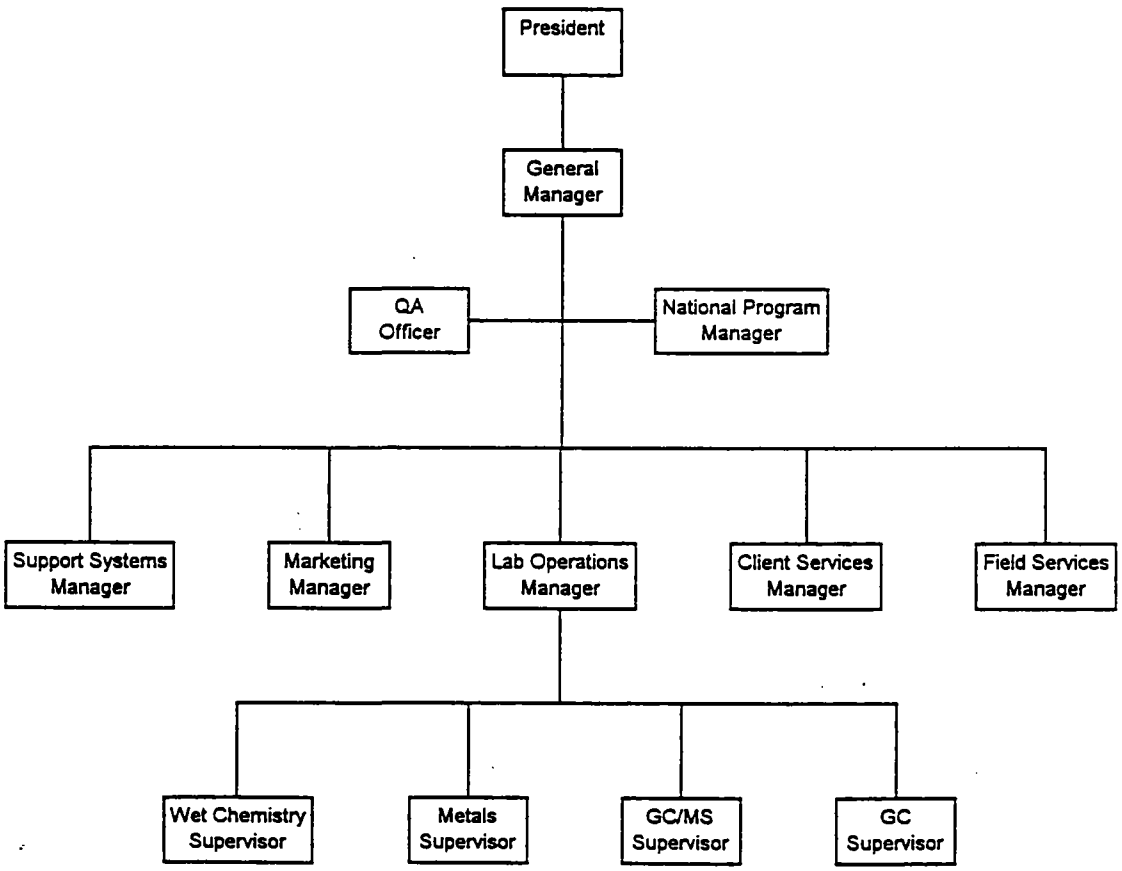


FIGURE 2-2

Pace Analytical Services, Inc. - Houston Laboratory Organization Chart



3. QUALITY ASSURANCE OBJECTIVES

3.1 INTRODUCTION

The purpose of this Quality Assurance Plan is to define procedures for the establishment of analytical systems and for the acquisition, documentation, evaluation, reporting, and archival of legally defensible data of known quality. The objective is to provide uniform systems for sample receipt, sample handling, instrument maintenance and calibration, methods control, performance evaluation, obtaining analytical data, evaluation of the quality of the data, and reporting. Specific procedures to be used for maintaining chain of custody, sample receipt and storage of samples, preventive maintenance, chemical analyses, internal quality control, reporting, QA audits, and corrective actions are described in specific sections of this plan or in standard operating procedures (SOPs) included by reference. This section addresses the objectives of accuracy, precision, completeness, representativeness, and comparability.

3.2 PRECISION AND ACCURACY

The QA objectives for precision and accuracy are to establish and maintain analytical systems that produce analysis results supported by QC data within acceptance criteria specified in the proposed analytical procedures. Precision and accuracy guidelines for the organic and inorganic procedures recommended by the USEPA are normally specified in the individual methods. These provide guidance as laboratory specific criteria are developed for each analytical method.

3.2.1 ORGANICS

Due to the extensive number of organic compounds selected as target analytes and environmental sample matrices, the development of precision and accuracy objectives and control limits for each analyte in all potential matrices is impractical. Thus, information is obtained for water and solid matrices that are representative of the normal environmental sample types. This is accomplished by determining the percent recovery of (1) matrix spike and matrix spike duplicate compounds added to selected samples before extraction and analysis, (2) surrogate spike compounds which are added to every sample, prior to extraction and analysis, and (3) laboratory control samples (LCSs), which are samples prepared from reference materials in DI water or clean sand and taken through the entire sample preparation and analysis procedure.

3.2.2 INORGANICS

Precision and accuracy data for inorganic test parameters are obtained by analyses of duplicate samples as a measure of precision and matrix spike and laboratory control samples as a measure of accuracy. At least one duplicate sample and matrix spike sample are analyzed per sample matrix type (e.g. water, soil) and concentration (e.g. low, medium) per batch of samples or for each 10 samples analyzed, whichever is more frequent, or as specified by project requirements. Samples identified as field blanks can not be used for matrix spike/duplicate sample analysis. If two analytical methods are used to obtain the reported values for the same element for a batch of samples (i.e. ICP, GFAA), matrix spike/duplicate samples are analyzed by each method. The matrix spike recovery and relative percent difference (RPD) of the duplicates for each component is calculated for data assessment. The method of standard additions (MSA) is employed, where applicable to the specific method performed, in order to achieve accuracy when matrix interferences are present in a sample.

3.2.3 FIELD SAMPLING

Field blanks and duplicates are collected and analyzed to assess field sampling activities. The analytical results for these samples provide data indicating procedural contamination, ambient conditions at the site, and representativeness of the analysis sample.

3.3 COMPLETENESS

Completeness is a measure of successfully obtaining all information necessary for a valid scientific study. The objective for completeness is: The methodology proposed for chemical characterization of the samples collected will provide data meeting QC acceptance criteria, following standard laboratory data review and validation, for at least 95% of all samples collected. Completeness may also be defined as a comparison of the number of tests successfully completed (with acceptable QC) to the number of tests requested. Nonconformance/corrective action (NC/CA) records are completed in accordance with standardized procedures in order to provide explanation when QC criteria are not met. The NC/CA record is completed by the analyst describing the situation encountered. The corrective action required is taken and documented in the appropriate section of the NC/CA record by the supervisor and analyst. See Section 13, Corrective Action.

3.4 REPRESENTATIVENESS

Representativeness is a qualitative measure that is related to the ability to obtain a sample that best reflects the characteristics of that part of the environment that is to be assessed. The laboratory utilizes homogenization of the sample, if compatible with the tests to be performed, to ensure the results obtained are representative of the sample as received.

3.5 COMPARABILITY

Comparability is also considered during preparation of the work plan. The objective of comparability is to produce results that do not differ significantly from those produced by other parties for the same purpose. Pace-Houston uses SOPs based on EPA-approved methods in order to achieve comparability with data from previous studies and from other laboratories. SOPs are written to incorporate the method requirements specified by the Pace corporate office in minimum requirements documents (MRDs), thus promoting comparability within the Pace system of laboratories. If an EPA-approved procedure is not available or not required for the analyte(s) or matrix to be analyzed, alternative published and/or validated procedures are submitted for client review and approval prior to analyses of samples.

Pace-Houston participates in external and inter-laboratory performance evaluation (PE) studies as an additional means of establishing comparability in the laboratory. Pace-Houston participates in USEPA Water Pollution (WP) and Water Supply (WS) studies, USEPA DMR-QA studies, and PE programs for various state and federal agencies and commercial clients. Pace-Houston also periodically analyzes single-blind QC check samples in order to internally monitor performance of parameters which are not evaluated by external PE studies.

4. SAMPLING PROCEDURES

4.1 PURPOSE AND APPLICABILITY

This procedure establishes requirements for the preservation of samples and the collection of samples by Field Services Department at Pace-Houston.

4.2 RESPONSIBILITIES

4.2.1 FIELD TECHNICIANS

Field Technicians are responsible for the collection of samples and for documentation of each sampling event in accordance with this procedure. Field Technicians are also responsible for field equipment decontamination and maintenance.

4.2.2 FIELD SUPERVISOR

The Field Supervisor is responsible for training field technicians and supervising their work, including review of sampling documentation and field reports. The supervisor is the on-site team leader for major projects.

4.2.3 FIELD MANAGER

The Field Manager is responsible for work plan and safety plan development. The Field Manager is also responsible for adherence to all quality assurance and regulatory guidelines.

4.2.4 SAMPLE CUSTODIANS

The Sample Custodians are responsible for storing samples and retrieving them from storage. Sample Custodians also add preservatives to sample containers, ship them to clients, and verify preservation of samples upon receipt at the laboratory.

4.2.5 SAMPLE COURIER

The sample courier is responsible for accepting samples under chain-of-custody from clients and samplers at field locations.

4.2.6 LABORATORY DEPARTMENT MANAGERS

The Laboratory Department Managers are responsible for scheduling sample analyses to ensure that holding times are met.

4.3 PROCEDURE

4.3.1 MATERIALS

a. Sample Containers

Sample containers are constructed of polyethylene or glass, as listed on Tables 4-1, 4-2, and 4-4. Pace-Houston routinely uses new sample containers obtained from a supplier that has been qualified, by the laboratory's analysis of DI water bottle blanks, to deliver containers free from contamination. Sample containers are used once only and disposed of according to federal, state, and local guidelines. In addition, Pace-Houston can supply certified, precleaned bottles upon request. Sample containers are stored in an area free of contamination.

b. Sample Preservation

Sample preservation techniques and holding times for non-CLP work are listed in the following tables:

- Table 4-1 – NPDES, NPDWR, and Aqueous RCRA Samples
- Table 4-2 – Non-Aqueous RCRA Samples
- Table 4-3 – Samples undergoing TCLP
- Table 4-4 – CLP Samples

The reagents and glassware used to prepare chemical preservatives are segregated and used only to prepare preservatives. To verify freedom from contamination, a preservative blank is analyzed each time a new manufacturer's lot of reagent is used to prepare the preservative. The blank is analyzed prior to use of the new preservative.

Preservatives are added directly to the sample containers. Labels indicating the type of preservative and the date of preparation are placed on eachsample container. A log of sample bottle preparation is maintained, which lists the type and number of bottles prepared, the preservatives used (including manufacturer and lot number of reagents), the date, and the preparer. Refer to Pace SOP number HO-P-002, *Sample Storage, Tracking, and Bottle Preparation*, for detailed procedures for sample container preparation.

c. Sample Bottle Orders

- Bottle orders specifying the following information are prepared by the Project Manager on a bottle order (Figure 4-1) or equivalent. Bottle orders must be placed as far in advance as possible.

  - Analyses, matrices, and number of samples
  - EPA program (RCRA, NPDES, NPDWR, etc.)
  - Name and address if bottles are to be delivered
  - Date required (this should be at least one day before sampling, so that if the client requires additional bottles, they can be sent).
  - Mode of transportation to the sampling site.

  The bottle order should also request chain-of-custody procedures (chain-of-custody record, Figure 4-2 or equivalent, and custody seals on coolers), trip blanks, etc. when required for the project.

- Trip blanks are prepared by completely filling a 40-mL VOA vial with organic-free reagent water, pouring the water down the side of the vial to minimize turbulence. The last few drops are gently poured into the vial so that surface tension holds the water in a convex meniscus. The vial is then capped. If air space is present, repeat the procedure. The vial is labeled as a trip blank. Preserved and unpreserved vials are prepared for each blank.

Preparation of field blanks is dependent on their function. The specific purpose of a field blank is discussed with the client when the order for bottles is placed. Preparation instructions are then specified by the Project Manager on bottle order or in a project-specific work plan or quality assurance plan.

- The bottle order is filled by a Sample Custodian. Bottles, with proper preservatives added, are packed in shipping containers in a manner that minimizes breakage. If the sampling event includes perishable parameters, the bottles must be packed in thermal coolers. A temperature blank bottle is added to each cooler to allow the Sample Custodian to accurately record the cooler temperature when samples are later received.

- A chain-of-custody form is sealed in a plastic bag and sent with each bottle order to encourage clients to complete the form and return it with the samples

The bottles may be picked up at the laboratory, delivered by Pace to the site, or delivered by a third party carrier.

4.3.2 SAMPLE COLLECTION BY FIELD SERVICES

a. Sampling Procedures

The sampling procedures used by Pace-Houston Field Services are outlined in the following manuals:

- Test Methods for Evaluating Solid Waste, Volume II: Field Manual Physical/Chemical Methods, SW-846, November 1986, and updates.

- Handbook for Monitoring Industrial Wastewater, Chapter 6, U.S. EPA Technology Transfer, August 1973.

- Handbook for Sampling and Sample Preservation of Water and Wastewater, EPA-600/4-82-029.

- Groundwater Monitoring Technical Enforcement Guidance Document, EPA, September 1986.

b. Field Notes

Field notes are documented in bound laboratory notebooks or on prenumbered field forms that are later bound into books. The following information is documented for each sampling event, at a minimum.

- For each event:
  - Location of sampling event.
  - Identification and calibration, where appropriate, of equipment used in sampling or in taking field measurements.
  - Weather conditions.
  - Description of anomalies at the site.
  - Signature of sample collector.
- For each sample:
  - Sample matrix (groundwater, wastewater, soil, sludge, oil, etc.).
  - Type of sample (composite or grab).
  - Location of the sampling point.
  - Date and time of sample collection.
  - Field measurements.

Field notes must be filed chronologically by site. Field notes will be reviewed for completeness and appropriateness by the Field Supervisor or senior Field Technician prior to report preparation.

c. Chain-of-Custody Record

A chain-of-custody record (Figure 4-2 or equivalent) should be completed for each sampling event to document sample custody from the time of collection through transfer of custody to the laboratory. At a minimum, the chain-of-custody record must contain the following information.

- Analyses required
- Type of sample bottle (e.g., metals, water chemistry, cyanide).
- Sample identification.
- Signature of collector.
- Date and time of sample collection.
- Signature and inclusive dates and times of possession for each person taking custody of the samples.

d. Field Measurements

When requested, the following measurements must be made in the field at the time of sample collection because of holding time limitations:

- Residual chlorine
- pH
- Dissolved oxygen
- Sulfite
- Temperature

e. Sample Delivery to the Laboratory

Samples must be delivered to the laboratory in a manner such that the characteristics of the sample are preserved and the analyses can be completed within the holding times.

4.3.3 SAMPLE PICK-UP BY PACE

At sample pick-up, the Pace driver requests a completed chain-of-custody record or release document with the samples. The driver signs and dates the chain-of-custody record or release document.

- Location of the sample pick-up (company and plant name).
- Date and time of pick-up.
- Driver's signature.

If the client does not provide a chain-of-custody record or release document, the driver completes a chain-of-custody record with the following information and obtains the signature (along with the date and time) of the client representative relinquishing custody of the samples:

- Client (company) name and location.
- Sample identification of each sample if they are not in sealed shipping containers. If the samples are sealed in shipping containers, the number of shipping containers is noted in the comments portion of the form.
- The driver also signs the form (along with the date and time) signifying receipt of the samples.

4.4 RECORDS

The following records are maintained in support of this procedure:

- Sampling notes
- Chain-of-custody records
- Field duplicates data
- Bottle preparation logs

FIGURE 4-1
BOTTLE ORDER FORM

pace INCORPORATED
ENVIRONMENTAL LABORATORIES

900 Gemini Avenue
Houston, TX 77058
713-488-1610
FAX: 713-488-4661

BOTTLE ORDER FORM

(ONE ORDER PER SHIPMENT)

Project Mgr: _____

SHIP TO: (SHIPPING ADDRESS ONLY)
COMPANY: _____
ADDRESS: _____
CITY: _____ STATE: ____ ZIP: ____
ATTN: _____
PHONE: _____
OTHER CONTACT: _____

DATE OF ORDER: _____
DUE DATE: _____
SPECIAL INSTRUCTIONS: _____

CHECKED BY: _____
DATE CHECKED: _____
CTNS IN SHIPMENT: _____
TOTAL WT: _____
SHIPPED VIA: _____
DATE SHIPPED: _____
AIR BILL: _____
BILL CLIENT.
AMOUNT: $ _____
SHIPPER'S INITIALS: _____

ALL BOTTLE ORDERS INCLUDE PACE CHAIN OF CUSTODY FORMS, BOTTLE LABELS, CUSTODY SEALS AND SAMPLING INSTRUCTIONS

Individual Bottles

Plastic w/ Poly-cone Caps

Water Chemistry	___qt	___pt
BOD	___qt	
Metals (w/ HNO_3)	___qt	___pt
(wo/ HNO_3)	___qt	___pt
Nitrogen (H_2SO_4)	___qt	___pt
COD (H_2SO_4)	___8 oz	___4 oz
TOC (H_2SO_4)	___8 oz	___4 oz
Cyanide (NaOH)	___qt	
Sulfide (NAOH, ZNCAC)	___pt	
Bacteria (Sterile $Na_2S_2O_3$)	___8 oz	
Radiochemistry (HNO_3)	___gal	

Amber Glass w/ Poly-cone Caps

Oil & Grease (HCL)	___qt	
TPH (HCL)	___qt	
Phenolics (H_2SO_4)	___qt	___pt

Glass w/ Aluminum & Teflon Lined Caps

TOX (H_2SO_4)(Amber)		___pt	___8 oz
Organics	___gal	___1/2 gal	___qt
POX (2 X 40 ml septum vial/pkg)		___pkg	
VOA (2 X 40 ml septum vial/pkg; w/HCL)		___pkg	
VOA (2 X 40 ml septum vial/pkg; wo/HCL)		___pkg	
VOA (3 X 40 ml septum vial/pkg; w/HCL)		___pkg	
Solids (wide mouth jar)		___qt	___pt
		___8 oz	___4 oz
Soil VOAs		___4 oz septum vial	

Miscellaneous

Empty Ice Chests	___
Ice Packs (freeze gels)	___
Temperature Blank	✓
Other (specify)	

Prepared Bottle Kits

NPDES Kits

Composite Sample
- ___ BOD (1 qt)
- ___ Cyanide (1 qt)
- ___ Phenolics (1 qt)
- ___ Organics (1 gal)
- ___ Nitrogen (1 qt)
- ___ Water Chem (1 qt)
- ___ Metals (1 qt) w/ HNO_3
- ___ Sulfide (1 pt)
- ___ Radiochem (1 gal)

Grab Sample
- ___ Bacteria (4 X 8 oz) ___ (8 oz)
- ___ Oil & Grease (4 X 2 qt) ___ (1 qt)
- ___ VOA (8 X (2 X 40 ml)) w/ HCL ___ (2 X 40 ml) w/ HCL
- ___ Field Blank (2 X 40 ml vials, filled with organic-free DI water)

Appendix IX Sampling Kit

- ___ Organics (3 X 1 gal)
- ___ VOA sets (3 X (2 X 40 ml))
- ___ Cyanide (1 qt)
- ___ Metals (1 qt)
- ___ Sulfide (1 pt)

Field & Trip Blanks

VOA Field Blank ___pkg ___w/ HCL ___wo/ HCL
(2 X 40 ml septum vials, empty; supplied w/ organic-free DI water)

VOA Trip Blank ___pkg ___w/ HCL ___wo/ HCL
(2 X 40 ml septum vials, filled w/ organic-free DI water)

Other (specify) _____

Waste Characterization Kit

- ___ TCLP & ZHE: (1 qt WM jar and 4 oz vial)
- ___ TCLP (only): 1 qt WM jar
- ___ Aqueous TCLP & ZHE: (1 gal & 2 X 40 ml vials wo/ HCL)
- ___ RCI: 1 qt WM jar

Sampling Kits

- ___ Transformer Oil (2 oz bottle)
- ___ PCB Wipe (4 oz metal can)
- ___ Stormwater grab ___ comp ___

WHITE PACE SHIPMENT RECORD
YELLOW CLIENT
PINK INCAN ORDER QUOTE SHEET

FIGURE 4-2
FIELD CHAIN-OF-CUSTODY RECORD

pace ANALYTICAL
ENVIRONMENTAL LABORATORIES

234331

CHAIN-OF-CUSTODY RECORD
Analytical Request

Client
Address
Phone
Report To:
Bill To:
P.O. # / Billing Reference
Project Name / No.
Pace Client No.
Pace Project Manager
Pace Project No.
*Requested Due Date:
Sampled By (PRINT):
Sampler Signature
Date Sampled

ITEM #	SAMPLE IDENTIFICATION	DATE	TIME	MATRIX	PACE I.D. NO.	NO. OF CONTAINERS	PRESERVATIVES: UNPRESERVED	H_2SO_4	HNO_3	HCl	NaOH	ANALYSES REQUEST	REMARKS
1													
2													
3													
4													
5													
6													
7													
8													

COOLER NOS.	SEALED?	SHIPMENT OUT DATE	METHOD / RETURNED DATE	ITEM #	RELINQUISHED BY / AFFILIATION	ACCEPTED BY / AFFILIATION	DATE	TIME

Additional Comments

SEE REVERSE SIDE FOR INSTRUCTIONS

TABLE 4-1

Container, Preservation and Holding Time Requirements Aqueous Samples - Non-CLP Work

Parameter	Container[1]	Preservative[2][5]	Maximum Holding Time[3]
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
BOD	P, G	Cool, 4°C	48 hours
Boron	P	HNO_3 to pH <2	6 months
Bromide	P, G	None	28 days
Chloride	P, G	None	28 days
Chlorine, Residual	P, G	None	Analyze Immediately
Chromium, Hexavalent	P, G	Cool, 4°C	24 hours
COD	P, G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH >12[4]	14 days
Dissolved Oxygen			
by Probe	G bottle and top	None	Analyze Immediately
by Winkler Titration	G bottle and top	Fix on site and store in dark	8 hours
Fluoride	P	None	28 days
Hardness	P, G	HNO_3 to pH <2	6 months
Iodide	P, G	Cool, 4°C	28 Days
MBAS	P, G	Cool, 4°C	48 hours

Parameter	Container[1]	Preservative[2][5]	Maximum Holding Time[3]
Mercury	P, G	HNO_3 to pH <2	28 days
Metals, except Boron, Cr^{6+}, and Mercury	P, G	HNO_3 to pH <2	6 months
Nitrogen			
Ammonia	P, G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Kjeldahl, Total	P, G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Nitrate-Nitrite	P, G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Nitrate	P, G	Cool, 4°C	48 hours
Nitrite	P, G	Cool, 4°C	48 hours
Odor	G, w/TLC	None	24 hours
Oil and Grease	G, w/TLC	Cool, 4°C; HCl to pH <2	28 days
Organic Carbon	P,G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Petroleum Hydrocarbons, Total	G, w/TLC	Cool, 4°C; HCl to pH <2	28 days
pH	P, G	None	Analyze Immediately
Phenolics	G, w/TLC	Cool 4°C; H_2SO_4 to pH <2	28 days
Phosphorus			
Elemental	G, w/TLC	Cool, 4°C	48 hours
Hydrolyzable	P, G	Cool, 4°C; H_2SO_4 to pH <2	48 hours

Parameter	Container[1]	Preservative[2][5]	Maximum Holding Time[3]
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Solids			
Dissolved	P, G	Cool, 4°C	7 days
Total	P, G	Cool, 4°C	7 days
Suspended	P, G	Cool, 4°C	7 days
Settleable	P, G	Cool, 4°C	48 hours
Volatile	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days
Sulfide	P, G	Cool, 4°C; Zinc acetate + NaOH to pH >9	7 days
Sulfite	P, G	None	Analyze Immediately
Temperature	P, G	None	Analyze Immediately
TOX	AG w/TLC	Cool, 4°C, H_2SO_4 to pH <2	28 days
Turbidity	P, G	Cool, 4°C	48 hours
BACTERIOLOGICAL TESTS			
Coliform, Fecal and Total	P, G sterile	Cool, 4°C[7]	6 hours
Fecal Streptococci	P, G sterile	Cool, 4°C[7]	6 hours

Parameter	Container[1]	Preservative[2][5]	Maximum Holding Time[3]
ORGANIC CHEMISTRY TESTS[6]			
Acrolein and Acrylonitrile	G with TLS	Cool, 4°C[7]; pH 4-5	14 days
Purgeable Halocarbons	G with TLS	Cool, 4°C[7]	14 days
Purgeable Aromatics	G with TLS	Cool, 4°C[7]; HCl to pH <2	14 days
Trihalomethanes/ VOCs	G with TLS[8]	Cool, 4°C; $Na_2S_2O_3$[9] (RCRA - HCl to pH <2)	14 days (NPDES - 7 days if not preserved with HCl)
Benzidines	G with TLC	Cool, 4°C[7]	7 days until extraction, 7 days after extraction
Chlorinated Hydrocarbons	G with TLC	Cool, 4°C	7 days until extraction, 40 days after extraction
Haloethers	G with TLC	Cool, 4°C[7]	7 days until extraction, 40 days after extraction
Nitroaromatics and Isophorone	G with TLC	Cool, 4°C[7]; store in dark	7 days until extraction, 40 days after extraction
Nitrosamines	G with TLC	Cool, 4°C[7]	7 days until extraction, 40 days after extraction
PCBs	G with TLC	Cool, 4°C	7 days until extraction, 40 days after extraction
Pesticides	G with TLC	Cool, 4°C; pH 5-9	7 days until extraction, 40 days after extraction
Phenols	G with TLC	Cool, 4°C[7]	7 days until extraction, 40 days after extraction
Phthalate Esters	G with TLC	Cool, 4°C	7 days until extraction, 40 days after extraction
Polynuclear Aromatic Hydrocarbons	G with TLC	Cool, 4°C[7]; store in dark	7 days until extraction, 40 days after extraction

Parameter	Container[1]	Preservative[2][5]	Maximum Holding Time[3]
TCDD	G with TLC	Cool, 4°C[7]	7 days until extraction, 40 days after extraction
Herbicides	G with TLC	Cool, 4°C	7 days until extraction, 40 days after extraction
RADIOCHEMISTRY TESTS			
Gamma Spectrometry	P, G	HNO_3 to pH <2	
Gross Alpha	P, G	HNO_3 to pH <2	6 months
Gross Beta	P, G	HNO_3 to pH <2	6 months
Iodine 131	P, G	None	3 weeks
Radium	P, G	HNO_3 to pH <2	6 months
Strontium	P, G	HNO_3 to pH <2	
Tritium	P, G[10]	None	
Uranium	P, G	HCl to pH <2	
AQUATIC TOXICITY TESTS			
Chronic Tests	P, G	Cool, 4°C	36 hours
Acute and Serial	P, G	Cool, 4°C	72 hours

TABLE 4-1 NOTES

1. AG - amber glass
   G - glass
   P - polyethylene
   TLC - teflon-lined cap
   TLS - teflon-lined septum

2. Sample preservation should be performed immediately upon sample collection. For composite samples, samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

3. The holding times listed are the maximum times that samples may be held before analysis and still be considered valid under EPA regulations. Holding times are measured from date of sampling.

4. Add 0.6 g ascorbic acid if residual chlorine is present.

5. If the dissolved content is to be measured, samples should be filtered on site immediately before adding preservatives.

6. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine (if present) with 0.008 percent sodium thiosulfate, storing int he dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are:

   - 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

   - Extracts may be stored up to 7 days before analysis for benzidines if storage is conducted under an inert (oxidant-free) atmosphere.

7. Add $Na_2S_2O_3$ if residual chlorine is present (0.008 percent).

8. Samples are to be collected in duplicate; provide two vials per sample. Also provide duplicate trip blanks for each sample set.

9. Omit $Na_2S_2O_3$ if maximum trihalomethane potential is to be determined.

10. New Jersey DEP requires that samples be collected in glass only for work performed in that state.

These requirements are based on 40 CFR 136, Table II; SW-846, Third Edition, Revision 2, Table 2-33; and EPA 814B-92-002, Table IV-4 and IV-5.

TABLE 4-2

Container, Preservation and Holding Time Requirements Non-Aqueous Samples - Non-CLP Work

Parameter	Container[1]	Preservative[2][6]	Maximum Holding Time[3]
Volatile Organics			
Concentrated Waste	8 oz. wide-mouth glass with TLC	None	14 days
Soil/Sediment	8 oz. wide-mouth glass with TLC	Cool, 4°C	14 days
Sludge	8 oz. wide-mouth glass with TLC	Cool, 4°C	14 days
Semi-Volatile Organics			
Concentrated Waste	8 oz. wide-mouth glass with TLC	None	14 days until extraction, 40 days thereafter
Soil/Sediment	8 oz. wide-mouth glass with TLC	Cool, 4°C	14 days until extraction, 40 days thereafter
Sludge	8 oz. wide-mouth glass with TLC	Cool, 4°C	14 days until extraction, 40 days thereafter
Metals			
All except mercury	8 oz. wide-mouth glass with TLC	Cool, 4°C	6 months
Mercury	8 oz. wide-mouth glass with TLC	Cool, 4°C	28 days

1. TLC = teflon-lined cap.

2. Soil/sediment and sludge samples should be cooled to 4°C for all parameters.

3. The holding times listed are the maximum times that samples can be held before analysis and still be considered valid under EPA regulations. Holding time is measured from sampling.

TABLE 4-3

Holding Time Requirements for Samples Undergoing Toxicity Characteristic Leaching Procedure (TCLP)

Analysis	From field collection to TCLP extraction	From TCLP extraction to preparative extraction	From preparative extraction to analysis	Total elapsed time
Volatiles	14 days	N/A	14 days	28 days
Semivolatiles	14 days	7 days	40 days	61 days
Mercury	28 days	N/A	28 days	56 days
Metals (ex. Hg)	180 days	N/A	180 days	360 days

TABLE 4-4

Container, Preservation and Holding Time Requirements CLP Work

Parameter	Container[1]	Volume	Preservative[2,3]	Maximum Holding Time[4]
Metals except Hg	Aqueous: P with PLC	1000 mL	HNO_3 to pH <2	6 months
	Non-Aqueous: G with TLC	4-32 oz.	Cool, 4°C	6 months
Hg	Aqueous: P with PLC	1000 mL	HNO_3 to pH <2	26 days
	Non-Aqueous: G with TLC	4-32 oz.	Cool, 4°C	26 days
Cyanide	Aqueous: P with PLC	1000 mL	NaOH to pH >12; Cool,4°C	12 days
	Non-Aqueous: G with TLC	4-32 oz.	Cool, 4°C	12 days
Volatile Organics	Aqueous: G with TLS	2-40 mL vials	Cool, 4°C; Dark	10 days
	Non-Aqueous: G with TLC	4 oz.	Cool, 4°C; Dark	10 days
Semi-volatile Organics, Pesticides/PCBs	Aqueous: AG with TLC	1/2 Gal.	Cool, 4°C; Dark	5 days until extraction[6]; 40 days thereafter
	Non-Aqueous: AG with TLC	8-16 oz.	Cool, 4°C; Dark	10 days until extraction[6]; 40 days thereafter

TABLE 4-4 NOTES

1. AG = Amber glass
   G = Glass
   P = Polyethylene
   PLC = Polyethylene-lined cap
   TLC = Teflon-lined cap
   TLS = Teflon-lined septa

2. Sample preservation should be performed immediately upon sample collection. For composite samples, samples may be preserved by maintaining at 4°C until compositing and sample splitting are complete.

3. If the dissolved content is to be measured, samples should be filtered on-site immediately before adding preservatives.

4. The holding times listed are the maximum times that samples may be held before analysis and still be considered valid under EPA regulations. Holding times are measured from the verified time of sample receipt (TVSR) at the laboratory. (Holding time in the field must be minimized when organics and/or cyanide are parameters of interest.)

5. If residual chlorine is present, all 0.6 g ascorbic acid.

6. Separatory and sonication extraction procedures must be completed within the holding time. Continuous extraction procedures (applicable to aqueous samples only) must be started within the holding time.

5. SAMPLE CUSTODY

5.1 SAMPLE RECEIPT AND VERIFICATION

All samples received by the Pace-Houston laboratory are accompanied by a chain-of-custody form completed by the client and/or sampler. Pace Analytical Services, Inc. provides chain-of-custody forms for all containers supplied to the client. Figure 4-2 shows the Pace chain-of-custody form. Clients choosing to utilize chain-of-custody forms other than those provided by Pace are responsible for ensuring all essential information is included on the form used.

Samples are received in accordance with the procedures set forth in Pace SOP number HO-P-001, *Sample Receipt and Log-in*. Shipping containers are inspected for custody seals and the condition is noted in the sample receipt log. The shipping containers are then opened and inspected for enclosed documentation. The temperature inside the shipping container is determined and recorded on the chain-of-custody form. The sample bottles are inspected for breakage and/or evidence of leakage. The sample bottle labels are inspected and compared to the chain-of-custody. The pH of preserved aqueous samples is verified as soon as possible after sample receipt.

The chain-of-custody is compared to the Project Alert Form provided by the Pace Project Manager. Any discrepancy noted is described on a nonconformance/ corrective action (NC/CA) record (see Figure 13-1) and the Pace Project Manager is notified immediately. The Project Manager is responsible for contacting the client and determining the corrective action required. The action taken is recorded on the NC/CA record and maintained in the project file.

5.2 SAMPLE LOG-IN

Samples are logged into the Pace-Houston Laboratory Information Management System (LIMS) in accordance with the LIMS User's Manual and Pace SOP number HO-P-001, *Sample Receipt and Log-in.*

Upon entry of all required sample tracking and analysis information into the LIMS, the information is reviewed by the Pace Project Manager for accuracy and completeness. Any errors or omissions are corrected at this time.

5.3 SAMPLES RECEIVED WITH NO PAPERWORK

Samples received with no paperwork are held in refrigerated storage in the sample receiving area until the proper instructions for analysis are received. The client is contacted immediately and the resolution of the problem is documented in the project file.

5.4 SAMPLE STORAGE

The sample containers are stored in designated refrigerators according to the type of analyses to be performed. Sample storage locations are listed in Pace SOP number HO-P-002, *Sample Storage, Tracking and Bottle Preparation.* Samples to be analyzed for volatile organic compounds are stored in separate refrigerators designated for volatile samples. Samples requiring strict internal chain-of-custody (direct tracking) are kept in locked storage areas. All refrigerators are monitored daily by a designated member of the Sample Custodian staff to ensure the temperature is maintained within a range of 2-6°C. Deviations from this temperature range are noted in the temperature logbook and corrective action is taken immediately to ensure the integrity of the samples.

5.5 SAMPLE/DATA ACCESS AND INTERNAL CHAIN-OF-CUSTODY

Pace follows standard operating procedures to assure the integrity of samples, prevention of degradation, and to prevent disclosure of data to unauthorized personnel. In order to ensure that this policy is maintained, the laboratory facilities are operated under controlled access. Only employees are allowed into the laboratory. Visitors must register upon arrival and are allowed access to the facility only with an escort.

Policies and procedures for maintaining internal chain-of-custody of samples are contained in Pace SOP number HO-P-002, *Sample Storage, Tracking, and Bottle Preparation.* Samples requiring strict internal chain-of-custody are signed in and out of locked storage areas by sample custodians and analysts.

5.6 SAMPLE DISPOSITION

Samples not totally consumed during analysis and any excess extracts or digestates generated during sample analysis are disposed of in accordance with local, state, and federal regulations. Specific disposal requirements are arranged with the client prior to receipt of samples. Client stipulated disposition requirements are to be set forth in the contract. Sample disposal is described in Pace SOP number HO-P-007, *Laboratory Waste Disposal and Sample Disposition.*

6. CALIBRATION PROCEDURES AND FREQUENCY

Analytical instruments and equipment used to obtain measurements or record data to be used for calculations of analytical results are calibrated at a frequency and in a manner such that accuracy and reproducibility are consistent with the manufacturer's specifications for proper instrument operation, and the calibration is in compliance with the analysis method requirements.

Laboratory measurements are based upon comparisons to results obtained for the analyses of reference standards analyzed by the same method. The results obtained for the analyses of calibration standards are used to prepare calibration curves or calculate calibration factors. The results of the sample analyses are quantified using either internal or external calibration techniques. Typically calibration is achieved by the analyses of five calibration standards at concentration levels set forth in the referenced method.

All instruments are calibrated using standard solutions of known concentrations. Where available, the standards are prepared from certified reference materials traceable to NIST or from reference materials whose concentration has been verified against NIST-traceable materials. Calibration standards are routinely verified for accuracy immediately following calibration and throughout the analytical sequence by using second-source standards. Thermometers and balances are calibrated annually using NIST-traceable thermometers and weights. Daily verification of balance calibration is described in Pace SOP number HO-P-003, *Calibration Verification of Laboratory Balances*.

Calibration standards are prepared from commercially available traceable stock standard solutions. The identity of the stock solution, preparation procedure, date, preparer, expiration date, and identity of the calibration standard are recorded in a standards preparation logbook. The entry is dated and signed by the preparation analyst.

The laboratory calibration procedures utilized must meet or exceed the method calibration criteria for all analyses performed. In the event that calibration criteria are not met, scientific justification to proceed with analysis is provided by the analyst. Supervisor and QAO approval must be obtained, or appropriate corrective action taken prior to analysis of samples. The calibration procedure in the method is followed for each specific analysis. Calibration procedures are documented on computer printouts, in the analysis logbook, and/or bench sheets where applicable.

Recalibration is performed at specified time intervals, when indicated by initial or continuing calibration verification procedures, or when required by the contract required method. Calibration procedures are method-specific; consult the appropriate Pace SOP for details regarding initial and continuing calibration.

7. ANALYTICAL PROCEDURES

7.1 PURPOSE AND APPLICABILITY

This section specifies the sources of analytical methods used by Pace-Houston and defines controls on standard operating procedures (SOPs), their content, approval for use, distribution, and revision. A list of preparation and analytical methods performed at Pace-Houston is included as Table 7-1.

7.2 RESPONSIBILITIES

7.2.1 LABORATORY DEPARTMENT MANAGERS

The Laboratory Department Managers shall select laboratory methods and prepare SOPs and any subsequent revisions according to this procedure. SOPs may be authored by a designee, but the Department Managers are ultimately responsible for their preparation and approval.

7.2.2 QUALITY ASSURANCE OFFICER

The Quality Assurance Officer shall review and approve SOPs for use at the laboratory. The QAO is also responsible for the distribution of controlled copies of the SOPs and for maintaining the associated documentation.

7.2.3 LABORATORY GENERAL MANAGER

The Laboratory General Manager shall review and approve SOPs. He/she has overall responsibility for all procedures performed at the laboratory.

7.3 PROCEDURE

7.3.1 METHOD SELECTION

Pace Analytical Services, Inc. will use EPA-approved methodology for the analysis of environmental samples whenever such methods are available. If the applicable agency has not specified an approved method, Pace Analytical Services, Inc. will select a recognized and validated method for use.

While SOPs may vary somewhat from one Pace laboratory to another, all laboratories performing a given analysis will use the same methodology to the extent that the instrumentation and predominant sample matrices at each facility allow.

If a client requests Pace-Houston to use a method developed by the client, the lab will do so, but only for the specified work. Such methods are subject to the controls listed below.

7.3.2 METHOD CONTENT

All analytical procedures will be performed according to a written SOP, incorporating specifics regarding Pace-Houston's quality control procedures, set-up and operation of Pace-Houston's instrumentation, etc. The SOP will address the following:

Purpose - List the property, analyte, or class of compounds measured by the method and summarize the procedure.

Application - Describe the sample matrices, working ranges, and situations to which the procedure applies.

Interferences - Describe those matrix components known to interfere in the analysis and methods for preventing or compensating for an interference when available.

Safety Issues - Describe protective equipment, known safety hazards, and precautions.

Sample Handling and Storage - Describe sample preservation and storage requirements.

Equipment - Describe the instruments, glassware, and other equipment applicable to the procedure.

Reagents - Describe reagents' and standards' concentration, grade, preparation, and use.

Procedure - Describe the sequence of activities to be performed. This should include calibration or standardization, sample pretreatment, sample analysis, calculations, reporting limits, quality control checks,

and special glassware cleaning procedures as appropriate to the method.

References - List the reference method(s) from which information was derived in preparing the method.

7.3.3 SOP REVIEW AND APPROVAL

All SOPs will be approved by the appropriate Laboratory Department Manager, the QA Officer, and the Laboratory General Manager.

7.3.4 SOP DISTRIBUTION

Controlled copies of the appropriate SOPs will be distributed to central files within each laboratory group. Additional controlled copies will be distributed upon request.

7.3.5 SOP REVISION

The first time an SOP is distributed, it is denoted as revision A. Subsequent revisions are denoted as revision B, C, etc.. Revisions must be approved and distributed in the same manner as the original method.

7.4 RECORDS

A revision history of lab SOPs, controlled copy distribution records, and master hard copies of all SOPs will be maintained by the QA Department in support of this procedure.

Table 7-1

Preparation Methods

Test	Water Method(s)	Soil/Waste Method(s)
Toxicity Characteristic Leaching Procedure	—	EPA 1311
Synthetic Precipitation Leaching Procedure	—	EPA 1312
Separatory Funnel Liquid-Liquid Extraction	EPA 3510B	—
Continuous Liquid-Liquid Extraction	EPA 3520B	—
Sonication Extraction	—	EPA 3550A
Waste Dilution		EPA 3580A
Purge and Trap	EPA 5030A	EPA 5030A
Florisil Column Cleanup	EPA 3620A	EPA 3620A
Gel-Permeation Cleanup	EPA 3640A	EPA 3640A
Sulfur Cleanup	EPA 3660A	EPA 3660A
Acid Digestion of Waters for Total Recoverable or Dissolved Metals (FAA/ICP)	EPA 4.1.4, 200.2, 3005A	—
Acid Digestion of Aqueous Samples and Extracts for Total Metals (FAA/ICP)	EPA 3010A	EPA 3010A
Acid Digestion of Aqueous Samples for Total Metals (GFAA)	EPA 4.1.3, 3020A	EPA 3020A
Acid Digestion of Sediments, Sludges, and Soils	—	EPA 3050A
Ammonia Distillation	EPA 350.2	EPA 350.2
Fluoride Distillation	EPA 340.1, SM 4500-F⁻ B	—
Cyanide Extraction	—	EPA 9013

Analytical Methods

Test	Water Method(s)	Soil/Waste Method(s)
Purgeable Halocarbons by GC	EPA 601[1], 8010B[1]	EPA 8010B[1]
Purgeable Aromatics by GC	EPA 602[1], 8020A[1]	EPA 8020A[1]
Phenols by GC	EPA 604[2], 8040A[2]	EPA 8040A[2]
Pesticides/PCBs by GC	EPA 505, 608[1], 8081, CLP SOW OLM01.0-1.8	EPA 8081, CLP SOW OLM01.0-1.8
PCBs by GC	EPA 505, 608[1], 8081	EPA 8081
Herbicides by GC	EPA 515.1, 8151	EPA 8151
Volatile Petroleum Hydrocarbons by GC	EPA 8015A[3]	EPA 8015A[3]
Semivolatile Petroleum Hydrocarbons by GC	EPA 8015A[3]	EPA 8015A[3]
Volatiles by GC/MS	EPA 524.2, 624[1], 8260A, CLP SOW OLM01.0-1.8	EPA 8260A, CLP SOW OLM01.0-1.8

Table 7-1

Analytical Methods (continued)

Test	Water Method(s)	Soil/Waste Method(s)
Base Neutral & Acid Extractables by GC/MS	EPA 625[1], 8270B CLP SOW OLM01.0-1.8	EPA 8270B, CLP SOW OLM01.0-1.8
Metals by ICP	EPA 200.7, 6010A, CLP SOW ILM02.0-2.1	EPA 6010A, CLP SOW ILM02.0-2.1
Metals by Graphite Furnace or Flame AA	EPA 200-Series, 7000-Series, CLP SOW ILM02.0-2.1	EPA 7000-Series, CLP SOW ILM02.0-2.1
Mercury by Cold Vapor AA	EPA 245.1, 7470A, CLP SOW ILM02.0-2.1	EPA 245.5, 7471A, CLP SOW ILM02.0-2.1
Acidity, Total	EPA 305.1, SM 2310	—
Alkalinity, Total	EPA 310.1, SM 2320 B	—
Ammonia	EPA 350.1	EPA 350.1
BOD	EPA 405.1, SM 5210 B	—
CBOD	SM 5210 B	—
Carbon, Total Organic	EPA 415.1, EPA 9060	EPA 9060, ASA 90-3 Walkley-Black[4]
Chloride	EPA 325.2,325.3, 9251, 9252A SM 4500-Cl⁻ C	—
Chlorine, Residual	EPA 330.5, SM 4500-Cl G	—
Chromium, Hexavalent	EPA 7196A, SM 3500-Cr D	—
COD	EPA 410.4, HACH 8000	EPA 410.1 Mod.
Color	SM 2120 B	—
Cyanide, Amenable to Chlorination	EPA 335.1, 9010A	—
Cyanide, Weak Acid Dissociable	SM 4500-CN I	—
Cyanide, Total	EPA 335.2, 9010A CLP SOW ILM02.0-2.1	EPA 9010A, CLP SOW ILM02.0-2.1
Cyanide, Reactive	—	EPA 7.3.3
Dissolved Oxygen	EPA 360.1, 360.2,	—
Fluoride, Total	EPA 340.2, SM 4500-F⁻ C	EPA 340.2, SM 4500-F⁻ C
Iodide	EPA 345.1	—
TOX	EPA 9020B	EPA 9020B
Hardness, Total	EPA 130.2, SM 2340 B	—

Table 7-1

Analytical Methods (continued)

Test	Water Method(s)	Soil/Waste Method(s)
Nitrate, Nitrate+Nitrite	EPA 353.2	—
Nitrite	EPA 354.1	—
Nitrogen, Total Kjeldahl	EPA 351.2	EPA 351.2
Oil and Grease	EPA 413.1, 413.2, 9070, SM 5520 B	EPA 9071A,
Paint Filter Liquids Test	EPA 9095	EPA 9095
Total Petroleum Hydrocarbons	EPA 418.1	EPA 3550A/418.1
pH	EPA 150.1, 9040B	EPA 9045C
Phenolics	EPA 420.1, 420.2	EPA 9066
Phosphorus, Total or Ortho-	EPA 365.2	EPA 365.2
Solids, Total	EPA 160.3, SM 2540 B	SM 2540 G
Solids, Total Dissolved	EPA 160.1, SM 2540 C	—
Solids, Total Suspended	EPA 160.2, SM 2540 D	—
Solids, Total Volatile	EPA 160.4, SM 2540 G	SM 2540 G
Solids, Settleable	EPA 160.5, SM 2540 F	—
Specific Conductance	EPA 120.1, 9050	—
Sulfate	EPA 375.4	EPA 9038
Sulfide, Total	EPA 376.1, SM 4500-S_2^- E	—
Sulfide, Reactive	—	EPA 7.3.4
Sulfite	EPA 377.1	—
Surfactants	EPA 425.1	—
Temperature	EPA 170.1	—
Turbidity	EPA 180.1	—
Waste Corrosivity	—	EPA 1110
Waste Ignitability	—	EPA 1010, 1020A
Fecal Coliform	SM 9222 D	—
Total Coliform	SM 9222 B	—
Heterotrophic Plate Count	SM 9215 B	—

Table 7-1

Analytical Methods
(continued)

Notes:

Methods designated "EPA" are from the following sources:

40 CFR Part 136, Code of Federal Regulations
EPA-600/4-79-20, Methods for Chemical Analysis of Water and Wastes, March 1983 Revision and updates
SW 846, Test Methods for Evaluating Solid Wastes, November 1986, Third Edition and updates
EPA/600/4-91/010, Methods for the Determination of Metals in Environmental Samples, June 1991
EPA/600/4-88/039, Methods for the Determination of Organic Compounds in Drinking Water, December 1988

'CLP SOW" - USEPA Contract Laboratory Program Statements of Work OLM01.1 and ILM02.0

SM" - American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 18th Edition

GC or GC/MS methods modified by the use of capillary columns.

GC method for phenols modified by the use of capillary columns and an ion trap detector.

GC Petroleum Hydrocarbons methods are derived from a modified SW-846 Method 8015A and from American Petroleum Institute Methods /API:AA4, Method for Determination of Gasoline Range Organics, and Q/API:AA5, Method for Determination of Diesel Range Organics.

Methods of Soils Analysis, American Society of Agronomy, 2nd Edition, 1982

8. DATA REDUCTION, VALIDATION AND REPORTING

8.1 PURPOSE AND APPLICABILITY

This section defines the Pace-Houston procedures for data collection, reduction, entry into the LIMS, validation, and reporting. Where procedures for CLP work and non-CLP work diverge, data handling is described for both.

All data is collected, reduced, entered, validated, and reported in accordance with this procedure unless an alternate scheme is outlined in a project-specific plan.

8.2 RESPONSIBILITIES

8.2.1 ANALYSTS

Analysts conduct data collection and reduction in accordance with this procedure.

8.2.2 LABORATORY SUPERVISORS

Laboratory Supervisors review and approve analytical data. This task may be delegated to experienced analysts.

8.2.3 LABORATORY DEPARTMENT MANAGERS

Laboratory Department Managers are responsible for approving sample data and laboratory analysis reports, compiling reports, and authorizing report delivery to the client.

8.2.4 PROJECT MANAGERS

Project Managers prepare cover letters to accompany the report and authorize delivery of the report by the scheduled due date.

8.2.5 QUALITY ASSURANCE DEPARTMENT

For larger or more complex projects, the QA Department may also review laboratory analysis reports and quality control data.

8.3 PROCEDURE

8.3.1 DATA COLLECTION

a. Sample Preparation and Analysis

Sample preparation and analytical activities are documented in sufficient detail to allow the analysis to be recreated. This includes the following, at a minimum. The information must be recorded in a laboratory notebook or on preprinted worksheets, or retrievable from instrument output.

- The analytical activity being performed (i.e., the specific analytical method or preparation method performed).
- The person(s) performing the activity and the date and time that the activity was initiated.
  - When more than one analyst works on an analytical run, each must be identified.
  - If an activity has discrete components that extend over more than one shift, the person performing each component and the date and time that each component is initiated are documented. For example, in suspended solids analysis, filtration of samples and determination of initial weight are performed on one day, and determination of final weight after drying is performed the following day. Thus the analysis breaks down into two discrete components. The analyst performing each component and the date and time that each component is initiated are documented in the analysis log.
- Instrument parameters, including instrument identification and settings. Instrument settings may be referenced to previous documentation of instrument parameters.
- The analytical sequence must be documented (i.e., the chronological order of analysis). The following data for each sample, standard, and QC check run in the analytical sequence must be recorded and/or retrievable from an instrument printout (quantitation report, printer tape, etc.).

Units for all variables are specified, preferably in column headings.

- Pace sample number.
- Client identification if CLP sample.
- QC sample type identification (prep blank, ms, etc.) if QC sample.
- Dilutions made prior to taking a sample aliquot (actual initial and final volumes, not a ratio).
- Sample aliquot/final volume.
- Cell size (colorimetric analyses).
- Instrument reading.
- Final result.
- Percent recovery, RPD, range, or percent difference of quality control checks.

- The calibration curve from which data are quantified, identified by instrument and date run, or by reference to a notebook and page number or a filename, if the initial calibration is not included in the analytical run.
- Identification of the source of standards used for calibration, calibration verification, lab control samples, and matrix spikes, usually by reference to a standards prep notebook and page number.
- Notes regarding any anomalies (e.g., change in color, formation of precipitate) or difficulties (e.g., instrument malfunction) encountered during analysis.
- The notebook identification number on each page.

- For CLP work, data for only one case may be recorded on a page. The sample delivery group is identified at the top of the page.

- Whenever additional pages must be added to a laboratory notebook, the attachment is described on the notebook page and the book and page number are recorded on the attachment. This allows the documents to be re-attached if they become separated.

b. Data Recording and Error Correction

All handwritten data must be recorded using indelible ink. When an error in any hardcopy documentation of data is corrected, the person making the correction draws a single line through the erroneous data so as not to obscure the original entry. He/she then writes his/her initials, the date, and the correct information, if applicable, adjacent to the error. An explanation of the change should also be included, either as a written comment or by using one of the following codes:

E1 - Misspelled or illegible entry
E2 - Mathematical error
E3 - Wrong data entered
E4 - Transposition or sequencing error
E5 - Transcription (copying) error
E6 - Procedural change
E7 - Wrong statement or conclusion
E8 - Unnecessary entry
E9 - Instrument error

8.3.2 DATA REDUCTION

a. Qualitative Identification

Qualitative identification of organic compounds is performed according to CLP Statement of Work guidelines. Second column confirmation by GC is performed upon request or when specified by the requested method.

b. Quantitation

The equations used to calculate final results are specified in the appropriate laboratory methods and SOPs. In general, the following rules concerning reporting limits, significant figures, and rounding rules apply to those calculations.

- Round all calculation results to the correct number of digits as the final calculation step. Do not round any result before reaching the final answer, even in a lengthy calculation.

- To round a number, first determine the number of digits to be reported (the reportable figures). Determine whether the digit to the immediate right of the right-most reportable figure is greater than, equal to, or less than 5. Ignore any digits further to the right unless the number is 5.

- If the number is greater than 5, round up (i,e., increase the right-most reportable figure to the next highest number).

  - If the number is less than 5, simply truncate after the last reportable figure.

  - If the number is equal to 5 and there are non-zero digits to the right of it, round up.

  - If the number is equal to 5 and there are no non-zero digits to the right of it, round up when the preceding figure is odd; truncate when the preceding figure is even.

- Round results at the end of calculations to one or two digits as follows, with the exceptions noted below.

  - If the initial concentration of the sample (i.e., the concentration before any dilutions are taken into account) is less than the reporting limit, express the reporting limit as 1 digit (e.g., <1 mg/L, not <1.0 mg/L).

  - If the initial concentration of the sample is above the reporting limit, and if expressed in scientific notation its exponent would be equal to that of the reporting limit

expressed in scientific notation, report the result to 1 digit. (For example, if the reporting limit is 1 mg/L, the initial concentration is 7.8965 mg/L and no dilutions were made, report the result as 8 mg/L.)

- If the initial concentration is above the reporting limit, and if expressed in scientific notation its exponent would be greater than that of the reporting limit expressed in scientific notation, report the result to 2 digits. (For example, if the reporting limit is 1 mg/L, and the initial concentration is 78.965 mg/L and no dilutions were made, report the result as 79 mg/L.)

Note: (1) For CLP work, report results in accordance with the statement of work, which may differ from these guidelines. (2) For quality control checks and PE samples, express results, recoveries, and relative percent differences using at least three significant figures whenever possible. (Additional reportable figures are not required for method blanks.)

- With some exceptions (organic wastes, low-solids sludges, etc.), solid samples are analyzed for percent moisture and reported on a dry weight basis. The reporting basis for solid samples should always be made clear in the analysis report.

c. Evaluation

The quality control data for each batch or analytical run are evaluated against acceptance limits. (See Section 9, Laboratory Quality Control.) Whenever a quality control result exceeds acceptance limits, with the exception of matrix spike recoveries, corrective action is required prior to turning in data for the batch or analytical run for independent data review. The client must be notified of any QC failure associated with their data and any steps taken to resolve the problem. Corrective actions are documented in the analysis log. When corrective action requires the efforts of someone other than the analyst, or the corrective action will not be completed during the work shift, the analyst completes a nonconformance/corrective action record. (See Section 13, Corrective Action.)

8.3.3 DATA ENTRY

After data reduction, the analyst enters the following data from the analytical run into the laboratory information management system (LIMS).

- Date and time of analysis (time initiated).
- Analyst.
- Instrument.
- Sample results.
- QC sample results.
- Lab notebook reference.

For CLP work, data is entered into the appropriate CLP forms generation software. Quality control data are entered into the LIMS. Sample results are simply entered into the LIMS as "done."

8.3.4 DATA VALIDATION AND REPORTING

The data validation and reporting sequence for CLP and non-CLP work are described below. The independent data and final report reviews conducted by the operations staff are intended to complement one another. The independent data review evaluates the results obtained for many samples for one test; the final report review evaluates the results for all tests run for one sample. Errors and inconsistencies that are not apparent from the initial review may be evident in the second review.

a. Independent Data Review - CLP and Non-CLP Work

- Following data reduction and before or after data entry, the raw data associated with the analytical run - analysis log, instrument output (quantitation reports, chromatograms, spectra, strip chart recordings), calibration curves, etc., are forwarded to the Laboratory Supervisor or his/her designee for independent review and approval. The review encompasses the correctness, acceptability, and completeness of the following elements of data generation and handling. (All elements are not applicable to all tests.)

- Data entry
- Instrument tuning
- Initial calibration
- Continuing calibration/calibration verification
- Calibration blanks
- Method or preparation blanks
- Surrogate and/or lab control sample recovery
- Qualitative identifications
- Quantitation, including units and reportable figures
- Precision of duplicates
- Recovery of matrix spikes
- Holding times
- Data qualifiers/comments

When an unacceptable calibration or quality control check is generated, the data reviewer ensures that appropriate corrective action was taken prior to approving the data. Any defects are corrected. Raw data and data entry are also corrected as necessary. If corrective action cannot be taken, the sample results are qualified appropriately.

- Upon approval of the data, the reviewer initials the lab notebook page(s), worksheet(s), or instrument printout, and indicates approval of the data in the LIMS, which allows the data to proceed to final report generation.

- Following independent data review, preliminary results may be provided to the client when necessary. The results must clearly be

labeled as being preliminary and subject to change upon completion of laboratory review.

b. Report Preparation - Non-CLP Work

- Following independent review, data are available for report preparation through the LIMS. Reports consist of a lab analysis report and a quality control report. The lab analysis report contains the following information:

  - Client name and address (including the person to whose attention the report is being sent).
  - Pace report number.
  - Report date.
  - Pace client number.
  - Pace vendor number (optional).
  - Pace work order number (optional).
  - Project name (optional).
  - Date sampled (when provided to Pace) and date of sample receipt for each sample.
  - Determination, result, and units for each analyte for each sample.
  - Any comments about the sample or results (e.g., presence of a matrix interference).

- If requested by the client, results from organics analyses which fall between the method detection limit and the reporting limit may be reported with a flag indicating that the result is an estimate.

- The quality control report contains the following information as applicable to the analytes:

- Supplemental information, including batch number, method reference, date and time of analysis, analyst, and instrument used for each determination.

- Surrogate standard recoveries.

- Laboratory control sample recoveries.

- Method blank results.

- Matrix spike and duplicate or matrix spike duplicate results.

If any quality control sample result does not meet the applicable acceptance criteria, a footnote or comment will be included with the result in order to explain the nonconformance and corrective action taken, if appropriate.

- The quality control report may be further supplemented with initial and continuing calibration data and/or raw data upon request.

- The report from the LIMS and any supplemental information is forwarded to the Laboratory Department Manager, who compiles and reviews the report, ensuring that all deliverables are included.

c. Final Report Review - Non-CLP Work

When all of the results for the parameters assigned to a sample have passed independent data review, they are evaluated by the Laboratory Department Managers. For some projects, the QA department may also perform a final report review. Errors and inconsistencies that were not evident in the initial review may become apparent when each result is evaluated in light of the results obtained for the other parameters. Specifically, the following are reviewed:

- Units and reportable figures.

- Interparametric relationships (e.g., TDS/conductance, TOC/BOD/COD, dissolved/total, anion-cation balance, where approriate).

- Reasonableness of results given the available information about the sample.

- Method references.

Any problems with the data must be corrected before the final report is approved.

The Project Manager reviews the final report in order to confirm consistency with the client's request for analyses to be performed. The Project Manager also evaluates the data for such findings as historical trends, required reporting limits, permit limits, etc.

d. Report Preparation - CLP Work

When a portion (VOA, BNA, Pesticides/PCBs, Inorganics) of the CLP data package is completed, the Laboratory Supervisor reviews it for technical accuracy and completeness.

Components of the data package are forwarded to the Laboratory Department Managers, who finalize the case narrative, compile the completed data package, and authorize its delivery to the client.

8.4 RECORDS

The following records are maintained in support of this procedure:

8.4.1 HARDCOPY RECORDS

- Analysis logs
- Standard and reagent preparation logs
- Instrument printouts
- Calibration curves
- Complete deliverables for each job, original and any revisions

8.4.2 ELECTRONIC RECORDS

- Records entered at data entry into the LIMS
- Records of data approval in the LIMS

9. LABORATORY QUALITY CONTROL

9.1 PURPOSE AND APPLICABILITY

This procedure provides an overview of the quality control (QC) measures used to assess and control analytical processes at Pace-Houston. Specific information on quality control checks for individual laboratory departments is provided in Pace SOPs for individual analysis methods.

9.2 RESPONSIBILITIES

9.2.1 QUALITY ASSURANCE DEPARTMENT

The Quality Assurance Department (QAD) shall establish and publish acceptance limits for quality control checks and assist laboratory personnel in updating variable limits annually, at a minimum.

9.2.2 LABORATORY ANALYSTS

Laboratory analysts shall compare the results of quality control checks to the published acceptance limits, and shall take appropriate corrective measures whenever acceptance limits are exceeded. Corrective measures shall be documented.

9.3 PROCEDURE

9.3.1 QUALITY CONTROL PROGRAMS

a. Daily Quality Control Program

NOTE: The following discussion of the daily quality control program is general in nature. The test-specific requirements of the methods, as outlined in Pace SOPs, supersede these general requirements. In addition, client- or project-specific QC requirements may supersede those specified in this QA Plan.

- The daily quality control program includes a variety of QC checks inserted into the analysis process by the analysts. These checks include instrument tuning or sensitivity checks, continuing calibration or calibration verification checks, lab control samples, and method blanks. Calibration verification standard, continuing calibration standard, and lab control sample results are calculated as percent recovery. Method blank results are evaluated for the presence/absence of laboratory contaminants. These quality control checks monitor the accuracy of the analytical procedure in the absence of matrix interferences. Decisions to accept or reject analytical results are based on these quality control results.

  Acceptance limits for these checks are taken from EPA methods or are established by Pace-Houston from actual QC data. If these checks fail to meet acceptance limits, corrective action is required prior to continuation of analysis and/or reporting of the data. The corrective action taken for each out-of-control event must be described in the analysis log and approved by the data reviewer. In addition, when the corrective action described in the applicable method is not effective in correcting the problem, or if out-of-control QC data must be reported due to insufficient sample remaining for re-analysis, expired hold time, etc., a nonconformance/corrective action record is completed. (See Section 13, Corrective Action.)

- In addition to these checks, 1 in 10 samples (1 in 20 samples for GC/MS analyses) of a similar matrix is analyzed in duplicate and as a matrix spike, or as duplicate matrix spikes, to evaluate matrix effects. Accuracy is calculated as percent recovery of the matrix spike. Precision is calculated as the relative percent difference (RPD) of duplicates or matrix spike duplicates. Precision is not quantified when one or both results are "less than" values.

  Acceptance limits for these checks are also taken from the EPA methods or are established internally by Pace-Houston. If matrix spike recovery fails to meet acceptance limits and the analytical system yielded acceptable results for calibration standards, lab control standards, and surrogate standards, corrective action is not required. The associated sample

results are qualified, however, to indicate the probable presence of a matrix interference. If precision acceptance criteria are exceeded, reanalysis of the duplicates and all of the positive samples in the batch is required, except in certain cases where the sample matrix may be heterogeneous and the sample aliquot size is small (e.g. TOC or TOX in soil). The corrective action taken is described in the analysis log and approved by the data reviewer. In addition, when the corrective action described in the applicable method is not effective in correcting the problem, or if out-of-control QC data must be reported due to insufficient sample remaining for re-analysis, expired hold time, etc., a nonconformance/corrective action record is completed. (See Section 13, Corrective Action.)

- Control limits for LCS and surrogate recoveries are statistically derived using historical laboratory data; these limits are updated annually, at a a minimum.. In addition to meeting the control limits and QC requirements specified in the method, the statistically-derived control limits are used to monitor performance of the method over time.

b. Performance Evaluation Studies

Pace-Houston participates in a variety of federal and state interlaboratory performance evaluation studies for drinking water, wastewater, and hazardous waste programs. Participation in these programs allows the lab to evaluate its performance against the performance of many other laboratories. Performance evaluation studies are also discussed in Section 10, Performance Evaluations and System Audits.

9.3.2 ACCEPTANCE LIMITS

Acceptance limits for the daily QC program are taken from EPA methods or are established by Pace-Houston from actual QC data as described in this section. Acceptance limits are calculated and summarized annually, at a minimum, and distributed to laboratory operations personnel by the QAD.

a. Fixed Limits

In general, acceptance limits for GC, GC/MS, and metals analyses for tuning, initial and continuing calibration, method blanks, and precision and accuracy of matrix spikes and duplicates or duplicate matrix spikes are based on acceptance limits established in EPA methods.

b. Variable Limits

Variable limits are based on laboratory-generated data and are updated annually, at a minimum.

- Accuracy

  Acceptance limits for percent recovery of lab control samples and GC and GC/MS surrogate standards are calculated from actual QC data. The mean (x) and standard deviation (s) are calculated from the most recently generated percent recovery data. A minimum of 20 values are necessary to establish limits; up to 100 values will be used when available. Outliers, which are excluded from the calculation of acceptance limits, are identified as described in paragraph 9.3.5.

  If the data generated are insufficient to calculate acceptance limits, and the method does not provide acceptance criteria, the following limits will apply:

  - Inorganic chemistry: 75.0 - 125% recovery
  - Metals: 75.0 - 125% recovery (water); 50.0 - 150% recovery (soils)
  - Organic chemistry - Volatiles: 75.0 - 125% recovery
  - Organic chemistry - Base-neutrals and other extractables: 50.0 - 150% recovery
  - Organic chemistry - Acids: 25.0 - 125% recovery.
  - Other analyses: 75.0 - 125% recovery

Acceptance limits are calculated as follows, where x_i represents the individual values and n is the number of values:

Parameter	Symbol	Formula
Upper Control Limit	UCL	$\bar{x} + 3s$
Upper Warning Limit	UWL	$\bar{x} + 2s$
Center Line (mean)	$\bar{x}$	$(\Sigma x_i)/n$
Lower Warning Limit	LWL	$\bar{x} - 2s$
Lower Control Limit	LCL	$\bar{x} - 3s$

- Precision

  Acceptable relative percent difference (RPD) of duplicate analyses is ≤ 20% for duplicate results greater than 10 times the method detection limit (MDL). When one or both results are ≤10 times the MDL, the RPD acceptance range is ≤ 67%. These limits are not applicable to cases where the sample matrix may be heterogeneous and the sample aliquot size is small (e.g. TOC or TOX in soil).

- Acceptance Limit Updates

  Acceptance limits will be updated annually at a minimum, when 20 or more new values have been generated. The summary of acceptance limits is revised and distributed to the appropriate lab groups after each update.

9.3.3 CONTROL CHARTS

In order to monitor each analysis for trends, laboratory control sample (LCS) recoveries are plotted on control charts (see Figure 9-1). Separate control charts are maintained for LCSs prepared in aqueous and solid matrices, where applicable. The LCS is the best indicator of a problem in the preparation step of many analyses. On tests which do not involve a distinct preparation step, as in volatile organics and several wet chemistry tests, an independent initial calibration verification (ICV) standard and/or continuing

calibration verification (CCV) standard from a second source is used to verify instrument and analyst performance, as well as initial calibration. This ICV or CCV is also designated an LCS for QC data reporting purposes.

9.3.4 OUT-OF-CONTROL SITUATIONS

Whenever an out-of-control situation occurs, with the exception of matrix spike recoveries (see 9.3.1.a.), corrective action is required prior to continuation of analysis and/or reporting of the data for the entire batch. The corrective action taken for each out of control event must be described in the analysis log. In addition, if out-of-control QC data must be reported due to insufficient sample remaining for re-analysis, expired hold time, etc., a nonconformance/corrective action record is completed. (See Section 13, Corrective Action.)

a. Fixed Limits

Analyses are out of control whenever the acceptance limits are exceeded.

b. Variable Limits

Analyses are out of control whenever the acceptance limits are exceeded. In addition, any of the following situations occurring on LCS control charts must be reported to the QAD. After evaluating the data, the QA Officer, along with the Laboratory Department Manager or Supervisor, will determine the required corrective action.

- 2 consecutive points are outside the warning limits
- 7 consecutive points are on one side of the center line
- 7 consecutive points increase or decrease
- An obvious cyclical pattern is observed in the distribution of points

9.3.5 IDENTIFICATION OF OUTLIERS

Outliers, which are excluded from the data used to calculate acceptance limits, are identified as follows (according to the procedure described in

ASTM E178-80, *Sections* 4.1 and 4.2):

- $\bar{x}$, s, and n are those values calculated from the current set of data.
- x_f is that value in the current set of data furthest from $\bar{x}$.
- $T_{1\%}$ is the critical value for T at the upper 1% significance level, corresponding to n, from Table I in ASTM E178-80.
- If the absolute value of $(\bar{x} - x_f)/s \leq T_{1\%}$, x_f is not an outlier. Use this value and all other values in the data set to calculate acceptance limits.
- If the absolute value of $(\bar{x} - x_f)/s > T_{1\%}$, x_f is an outlier. Delete this value from the data set and do not use it to update acceptance limits. Repeat the test for outliers, using the new x, s, n, and x_f from the current data set, until no outliers are identified. More values from historical data may be added to the data set if n becomes less than 20.

9.3.6 METHOD DETECTION LIMITS

Method detection limits (MDLs) are determined for each analyte on each instrument by performing MDL studies as specified in 40 CFR Part 134, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit*, Revision 1.11. MDLs are updated annually, at a minimum.

9.3.7 REPORTING LIMITS / PRACTICAL QUANTITATION LIMITS

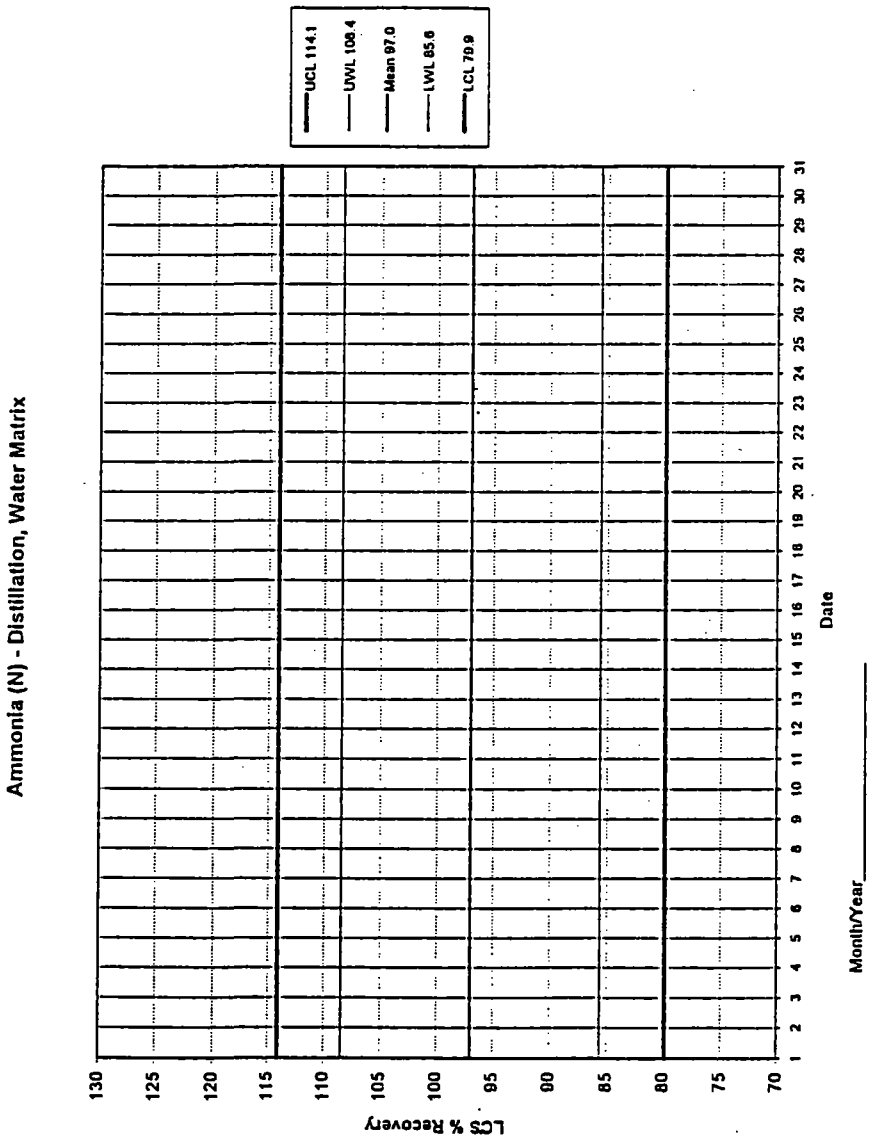
The reporting limit, or practical quantitation limit (PQL), for a given parameter is a nominally chosen value which can routinely be detected and reported as greater than zero in the majority of sample matrices encountered in the laboratory. Reporting limits are always greater than the corresponding MDLs and are established based upon the noise level of the instrumentation, experience in performing the analysis, and typical reporting limits required by regulatory agencies and clients. A reporting limit for a given parameter may vary according to the needs of the client.

9.4 RECORDS

All quality control data and a history of acceptance limits will be maintained by the Quality Assurance Department in support of this procedure.

Figure 9-1

LCS Control Chart



10. PERFORMANCE EVALUATIONS AND SYSTEM AUDITS

10.1 INTERNAL AUDITS

The records, systems, and procedures of each laboratory department are audited annually, at a minimum, by the Quality Assurance Department. The data is reviewed for completeness, accuracy, and adherence to standard operating procedures. Random project files are evaluated for compliance to procedures throughout the analytical process (i.e., from sample receipt through the final report). Department Managers and Supervisors check all logbooks and records to ensure appropriate documentation of analyses are being recorded in the proper manner.

10.2 EXTERNAL AUDITS

Pace-Houston is audited as required by regulatory agencies to maintain laboratory certifications and approvals. Commercial clients with laboratory auditing programs typically conduct on-site audits at least once per year, and perform data audits on a project-specific basis. These audits are conducted by the client or a consulting firm specializing in this service and operating under contract to the client.

10.3 PERFORMANCE EVALUATIONS

Pace-Houston participates in the US EPA semi-annual drinking water (WS Series) and semi-annual wastewater (WP Series) Performance Evaluation Studies (four studies per year). The laboratory also participates in various client-sponsored performance evaluations by analyzing QC samples prepared and submitted by commercial clients in conjunction with their own QA program. In addition, the Pace-Houston Quality Assurance Department periodically submits to the laboratory single-blind QC check samples, in order to internally monitor performance of parameters which are not evaluated by external PE programs.

11. PREVENTIVE MAINTENANCE

Pace-Houston employs a full-time instrument specialist to perform maintenance and repairs of most analytical instruments. Maintenance and repair of some instruments, such as analytical balances, is performed under service contracts with outside vendors. A list of major instruments is included as Table 11-1.

All instruments and equipment receive routine preventive maintenance, which is recorded in instrument specific maintenance logs. Routine maintenance ensures that the equipment is operating under optimum conditions, reducing the possibility of instrument malfunction.

Preventative maintenance procedures including lubrication, source cleaning, detector cleaning, and the frequency of such maintenance are performed according to the procedures recommended in the manufacturer's instrument user manual.

Chromatographic carrier gas purification traps, injector liners, and injector septa are cleaned or replaced on a regular basis. Precision and accuracy data are examined for trends and excursions beyond control limits to determine evidence of instrument malfunction. Maintenance must be performed when the instrument begins to degrade as evidenced by the degradation of peak resolution, shift in calibration curves, decreased sensitivity, or failure to meet one or another of the quality control criteria. Instrument logbooks containing maintenance and repair records are kept in the laboratories at all times. The laboratories also maintain adequate supplies of spare parts such as GC columns, syringes, septa, injection port liners, and electronic parts to minimize potential down-time.

In the event of equipment malfunction that cannot be readily resolved by laboratory personnel, service is obtained from the instrument vendor or manufacturer. Should instrument failure preclude completion of analyses within contract requirements (i.e., holding times), Pace-Houston will contact the client to determine alternative strategies, including use of another Pace Analytical Services laboratory.

Table 11-1

Pace-Houston Laboratory Major Instruments

Type		Make & Model	Year of Purchase
GC/MS	(3)	Finnigan INCOS 50	1986, 1987, 1987
GC/MS	(2)	Finnigan INCOS 500	1990, 1992
GC/MS		Finnigan 5100	1990
GC/MS	(2)	Finnigan 4500	1986, 1990
GC/MS	(2)	Hewlett Packard 5972	1994, 1994
GC/MS		Hewlett Packard 5971A	1995
GC w/ECD	(3)	Varian 3400	1987, 1987, 1991
GC w/ECD		Hewlett Packard 5890-II	1990
GC w/dual ECD	(2)	Hewlett Packard 5880A	1984, 1984
GC w/FID		Varian 3400	1989
GC w/FID		Hewlett Packard 5890	1980
GC w/PID	(2)	Hewlett Packard 5890-II	1991, 1991
GC w/PID	(2)	Varian 3300	1987, 1987
GC w/Ion Trap		Finnigan Magnum	1993
GPC Unit		ABC Labs 1002B	1988
ICP Spectrophotometer		Perkin-Elmer P1000	1991
ICP Spectrophotometer		Perkin-Elmer P40	1989
ICP Spectrophotometer		Thermo-Jarrell Ash 61E	1993
ICP Spectrophotometer		Thermo-Jarrell Ash 61E Super Trace	1994
Graphite Furnace AA Spect.		Perkin-Elmer 3030	1987
Graphite Furnace AA Spect.		Perkin-Elmer 5100	1991
Graphite Furnace AA Spect.		Perkin-Elmer 5100P	1994
Graphite Furnace AA Spect.		Perkin-Elmer 4100ZL	1990
Flame AA Spectrophotometer		Perkin-Elmer 3030	1986
CVAA Mercury Analyzer		Perkin-Elmer FIMS	1994
CVAA Mercury Analyzer		Bacharach 50B	1993
Autoanalyzer	(2)	Alpkem RFA300	1988, 1993
Autoanalyzer		Bran & Lubbee TRAACS-800	1988
TOC Analyzer		Dohrmann DC-80	1987
TOC Analyzer		Shimadzu ASI 5000 S	1991

Table 11-1
(continued)
Pace-Houston Laboratory Major Instruments

Type		Make & Model	Year of Purchase
TOX Analyzer		Mitsubishi TOX-10 Sigma	1995
TOX Analyzer		Dohrmann DX20	1986
TOX Analyzer		Mitsubishi TOX-10+	1988
IR Spectrophotometer		Perkin-Elmer 1310	1989
UV-Vis Spectrophotometer		Milton Roy 301	1988
UV-Vis Spectrophotometer	(2)	Milton Roy 401	1992, 1994

12. ASSESSMENT OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

12.1 ACCURACY

Accuracy is indicated by the measure of the difference between observed and true values. A minimum of one of every 20 environmental samples for organic analyses or one in 10 for inorganic analyses is spiked with a standard solution to assist in evaluating the accuracy of the method for a given sample matrix through calculation of percent recovery of the matrix spike.

Each batch of up to 20 samples is prepared with a laboratory control sample (LCS) to ensure the analysis system is operating in control. The percent recovery for the LCS is calculated by comparison of the value obtained for the analysis with the true value for the LCS.

Surrogate compounds are spiked into samples analyzed by GC and GC/MS methods. The percent recoveries of the surrogates are used as an indicator of the accuracy of the analysis.

The calculation of percent recovery is performed in the following manner:

- Matrix Spike Recovery -

$$\% \text{ Recovery} = \frac{SSR - USR}{SA} \times 100\%$$

where:
SSR = Spiked sample result
USR = Unspiked sample result
SA = Spike added

- Surrogate or Lab Control Sample Recovery -

$$\% \text{ Recovery} = \frac{\text{Result obtained}}{\text{True value}} \times 100\%$$

See Section 9, Laboratory Quality Control, for a discussion of control limits and corrective action for out-of-control events.

12.2 PRECISION

Precision refers to the reproducibility of results obtained for the analyses of duplicate samples or matrix spiked duplicate samples. One out of every 20 samples of similar matrix analyzed by each method for organics (1 in 10 for inorganics) is run in duplicate or as matrix spike duplicates for determining precision.

The results of the duplicate analyses are computed and the absolute relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{(R1 - R2)}{\frac{1}{2}(R1 + R2)} \times 100\%$$

where: R1 = First replicate result
R2 = Second replicate result

The RPD must fall within set acceptance limits for the results to be accepted and subsequent data validated. See Section 9, Laboratory Quality Control, for a discussion of control limits and corrective action for out-of-control events.

12.3 COMPLETENESS

Data completeness can be quantified during data assessment. A statement of expected completeness for a project is one data quality objective. Pace-Houston has established the ability to provide data, meeting QC acceptance criteria, for 95% or more of the requested determinations. It is incumbent for planners to identify any sample types, such as control or background locations, which require 100% completeness.

12.4 REPRESENTATIVENESS

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. It is specifically addressed in each work plan. The laboratory recognizes its role in achieving within-sample representativeness and uses appropriate techniques to obtain a representative aliquot. Some test are performed in quadruplicate if obtaining a representative aliquot is precluded by sample heterogeneity and small aliquot size.

12.5 COMPARABILITY

The objective of comparability is to produce results that do not differ significantly from those produced by other parties for the same purpose. Pace-Houston uses SOPs based on EPA-approved methods in order to achieve comparability with data from previous studies and from other laboratories. SOPs are written to incorporate the method requirements specified by the Pace corporate office in minimum requirements documents (MRDs), thus promoting comparability within the Pace network of laboratories. Pace-Houston participates in external and inter-laboratory performance evaluation (PE) studies as an additional means of establishing comparability in the laboratory.

13. CORRECTIVE ACTION

For purposes of the Pace-Houston corrective action system, nonconformances are defined as:

- Deviations from methods, SOPs, client requirements, internal QA/QC requirements, or good lab practices, which may have an effect on data or the client's interpretation of data, or

- Potential quality problems identified by PE samples, external clients or agencies, or Pace-Houston employees.

- Complaints, defined as expressed dissatisfaction with data or procedures related to data quality, from clients or other parties.

In order to identify, track, and prevent the recurrence of problems, nonconformances and the corrective actions taken are documented on a corrective action record (see Figure 13-1).

The steps that may comprise a closed-loop corrective action system are as follows:

1. Define the problem.

2. Assign responsibilities for problem investigation.

3. Communicate the problem to affected manager(s) and project management, where applicable.

4. Investigate and determine the cause of the problem (check all calculations, re-analyze the sample, verify the integrity of the spiking solution, laboratory control sample, or calibration standard, check instrument and operating conditions to preclude the possibility of malfunctions or operator error, etc.).

5. Determine the corrective action(s) necessary to eliminate the problem and to prevent its recurrence.

6. Assign and accept responsibilities for implementing the corrective action.

7. Report the nonconformance and corrective action taken to the Quality Assurance Department.

8. The completed corrective action record is closed by the QA Department and copies routed to the originator, the client file, and the Laboratory General Manger.

If investigation of a complaint reveals a systematic nonconformance to the QA program, an internal audit will be conducted by the QA Department to determine the scope of the problem, any affected data, and corrective action needed to prevent recurrence. If, as a result of the audit, a client's data are shown to be incorrect, the client will be immediately notified in writing and a corrected report delivered, when appropriate.

Figure 13-1

Nonconformance/Corrective Action Record

95-O-

Pace Analytical Services, Inc. - Houston Laboratory
NONCONFORMANCE / CORRECTIVE ACTION RECORD
See instructions on reverse side.

Originator: _____ Date: _____
Client: _____ Affected sample nos.: _____
Test: _____ Book & page no.: _____

Sample Integrity	Sample Analysis	External Origin
Route to PM, contact client ASAP	Take corrective action in lab, comment on final report	Take corrective action in lab, reissue final report when appropriate
__Preservative __Other __Temperature __VOA headspace __Container __ID/labeling __Holding time __Sampling	__LCS recovery __Other __Blank contamination __Calibration __Compound ID, qualitative __Calculation/quantitation __SOP/method deviation	__PE results __Other __Client problem/request __Agency requirement

Nonconforming condition: _____

ROUTE TO: _____ (Responsible individual) COPY TO: _____ (Affected manager) _____ (Project manager)

Corrective action (temporary fix): _____

By: _____ Date completed: _____

Preventive action (permanent fix): _____

By: _____ Date completed: _____

ROUTE ORIGINAL TO QA DEPARTMENT

Additional comments: _____

Closed by: _____ Date: _____

COPY TO ORIGINATOR, CLIENT FILE, AND GENERAL MANAGER

14. QUALITY ASSURANCE REPORTS TO MANAGEMENT

14.1 OBJECTIVE

This section describes the methods used by Pace-Houston to ensure that management personnel are informed of situations which could affect the performance of the laboratory.

14.2 PROCEDURE

Quarterly reports are provided by the Quality Assurance Officer to the Corporate Director of Quality and the Laboratory General Manager. This report addresses the quarterly quality assurance activities including details of corrective actions implemented, audit results, performance evaluation results, and other major quality issues when they arise.

In addition to the quarterly QA reports, weekly meetings are used to communicate to the laboratory's management staff pertinent information related to QA/QC issues.

15. TRAINING

15.1 INTRODUCTION

This section of the QA Plan describes the Pace-Houston program for training in areas where quality is affected. Specific areas where training must be documented include:

- Analytical methods training
- Quality assurance/quality control (QA/QC) training
- Safety training

Other types of training also occur but are not at this time required to meet the requirements of the quality assurance program; these include such subjects as computer training, continuing education, seminars, and total quality management (TQM) training.

Specific details of the training program at Pace-Houston can be found in Pace SOP number HO-P-004, *Training Procedures*.

15.2 ANALYTICAL METHODS TRAINING

All analysts are trained and supervised in performing specific analytical procedures before working unsupervised. The Laboratory Department Managers are responsible for training within their work groups. A supervisor or senior analyst typically conducts the training, using method-specific analytical SOPs as training guides.

A training record is used to document the trainee's proficiency in performing the procedure. For some methods, analyst proficiency is also demonstrated through the analysis of standard materials, with documentation retrievable from the lab notebook and raw data.

Each Department Manager will determine the frequency of retraining, based on revisions to the SOPs or the methods themselves.

15.3 QA/QC TRAINING

The Quality Assurance Officer (QAO) conducts training of new hires in general QA/QC principles. The QAO determines the frequency of retraining, based on deficiencies determined during performance evaluation or systems audits. Additionally, the QAO and/or Project Manager may provide project-specific training before the laboratory analyzes samples for a major project or a project with specific QA/QC or analytical requirements.

A training record is used to document each trainee's attendance at a given training session.

15.4 SAFETY TRAINING

The laboratory Safety Officer conducts training of new hires in the Pace safety program and Chemical Hygiene Plan, and in hazard communications (HAZCOM). Annually all employees are given safety training, which includes the following subjects:

- Safety and Chemical Hygiene Plan
- HAZCOM
- Blood-borne pathogens
- Fire safety

In addition, selected employees are trained annually in respirator use, waste handling/hazardous materials (HAZMAT), and/or first aid and CPR. The Safety Officer also conducts other safety-related training as needs arise. A training record is used to document each trainee's attendance at a given training session.

15.5 TRAINING RECORDS

Records are maintained documenting each employee's training in analytical methods, QA/QC principles, and safety. Training records shall specify the trainee, trainer, date, and subject of the training session. The results of proficiency testing, where applicable, should also be included.

16. PROCUREMENT AND CONTROL OF MATERIALS AND SERVICES

16.1 INTRODUCTION

This section of the QA Plan describes the policies and procedures for the control of purchased materials and services which affect quality. The procurement of materials and services must be preceded by a planning phase, in which the requirements of specific EPA methods, projects, or contracts are evaluated. This includes defining the acceptance criteria for standards, reagents, glassware, instrumentation, and subcontracted laboratory services. Detailed procedures for the procurement of materials and services are found in Pace SOP number HO-P-005, *Control of Purchased Items and Services.*

16.2 LABORATORY MATERIALS

16.2.1 PROCUREMENT

In planning for procurement of materials, acceptance criteria must be defined before selection of a supplier. The supplier should be provide evidence that the materials meet the acceptance criteria before being put into use. General guidelines for environmental laboratory materials include the following:

- Standards - calibration standards should be NIST-traceable, if available. Use a second source (manufacturer different from calibration standards) for calibration verification standards.

- Reagents and chemicals - use grade specified in method (i.e., reagent grade conc. HCl, purge and trap grade methanol).

- Volumetric glassware - use "Class A" only.

- Weights used to verify balance calibration - use "Class S" only. Certificates of calibration should be supplied.

- Instrumentation - use equipment described in the method, with adequate sensitivity, working range, and reliability. Certificates of calibration should be supplied for such items as millivolt standards

and NIST-traceable thermometers, where the item will be used to calibrate other equipment in the laboratory.

16.2.2 MATERIALS CONTROL

In order to ensure that deteriorated reagents and chemicals are not used, a label is affixed to each container when received at the laboratory, prompting for date received, date opened, and expiration date. Receiving personnel complete the blank for date received; when first opening the container, the analyst completes the label by supplying the date opened and expiration date. If the manufacturer has not specified an expiration date, the analyst completes the blank with "None".

Solvents used for organic extractions are prescreened for contamination by the laboratory whenever a new lot is received. If analysis of the solvent reveals a contaminant, the lot is rejected. If the solvent contains no detectable contaminants, the lot may be reserved by the supplier in large quantities for use by Pace-Houston. In addition, preservatives used in sample containers are prescreened whenever a new lot is received.

Reagents and chemicals are stored in appropriate containers, with incompatible materials segregated (see the Pace Analytical Services, Inc. Chemical Hygiene Plan). Acids are stored with the containers in separate plastic trays. Solvents are stored in designated solvent cabinets. Standards and working reagents are stored in refrigerators maintained at 2-6° C or in freezers, where appropriate.

Preparation of reagents and standards (calibration, calibration verification, surrogate spike, and matrix spike) is documented in support of sample analysis in a lab notebook as follows:

- Compound or element name and/or formula and final concentration or normality.

- Identification number, which takes the following form:

  999-99-888-7

  where "999-99" and "888" are the notebook number and page number of the entry, respectively, and "7" is the number of the entry on the standards notebook page.

- Manufacturer and lot number of all standard materials.
- Date prepared and preparer.
- Expiration date.
- Brief description of preparation. This may be referenced from a previous description of preparation or the lab method, providing that the standard or reagent was prepared in exactly the same fashion as the referenced description.

Each newly-received lot of calibration standards must be verified against the standard currently in use before being used for calibration in the laboratory. Procedures and criteria for standard verification are found in Pace SOP number HO-P-009, *Standard Verification*.

Bottles or flasks containing standards or reagents must be labeled with the following:

- Compound or element name and/or formula, and final concentration or normality.
- Identification number.
- Lot number
- Date prepared and preparer.
- Expiration date.

16.3 LABORATORY SERVICES

With prior consent by the client, Pace-Houston may use other laboratories in the Pace network to perform analyses not within Pace-Houston's capabilities or capacity. Subcontracted laboratory services (i.e., services of non-Pace laboratories) are also occasionally used by Pace-Houston with the client's consent.

When procuring subcontracted laboratory services, the minimum criteria to be met by the subcontracted laboratory must be established. The following should be addressed, at a minimum:

- Methods to be performed
- State or federal certifications or approvals required
- Special quality control requirements specified by project
- Report deliverables required
- Financial responsibility and insurance
- Contractual flowdowns
- Client's consent - blanket, or case-specific

Once a potential candidate lab is selected, the lab's quality assurance plan is reviewed by the Pace Quality Assurance Officer (QAO). A laboratory may not be used for subcontracted analyses without prior approval by the QAO. If the QA Plan is not available or appears inadequate, the QAO may schedule an audit of the candidate laboratory's facility.

In order to monitor the subcontracted laboratory's performance on an ongoing basis, the QAO shall periodically review subcontractors' analysis results and QC data.